

## **Summary of Research Report**

**Grant No. NAG-1-01054**

### **Accelerated Testing of Polymeric Composites: Correlation of Scale-up Effects on Viscoelastic Behavior**

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## **INTRODUCTION**

A major issue for many designers and engineers is the long-term mechanical properties of polymer matrix composites (PMC). These composites are being used more than ever in high-performance applications; and engineers need to understand the characteristics of polymeric behavior as they relate to the time-dependent mechanical properties, or viscoelastic properties. The viscoelastic nature of polymers dominates the mechanical properties of polymeric composites. Failure modes such as excessive deformation, creep rupture, and environmental aging are all related to the viscoelastic properties of these composites. Determining these time-dependent properties, and the factors that affect them, is crucial to understanding how a material will perform under high performance conditions.

In this research project an accelerated method was developed using the dynamic mechanical analyzer (DMA) to measure the viscoelastic creep properties of polymeric composite materials. The objectives of the study are to: investigate the use of the DMA in finding creep properties of polymeric composites, compare results from DMA creep tests with data from conventional creep tests, assess the accuracy of results of the DMA sub-coupon level tests, and investigate the potential of using the DMA results to predict the long-term creep properties of composite materials.

## **METHODOLOGY AND FINDINGS**

Initially a study was performed to prove the concept of using the DMA to predict long term creep behavior of polymer matrix composites. In this study flexure creep tests were performed on IM7/K3B composite material, and the results were compared with tensile

and compressive creep tests on the same material. The effect of physical aging of the material was not considered in this proof of concept study. A comparison of results from three test programs indicated that the loading mode caused a slight difference in creep behavior of polymer matrix composites. It was shown that while the DMA flexure creep results were not identical to the tension and compression creep results, the DMA test closely resembled the tension and compression creep behavior of polymeric matrix composites. Furthermore, the results confirmed a previous finding that the slight difference between flexure, tension and compression creep may reflect a real change in material behavior under these modes of loading. In spite of the combined loading modes (tension, compression and shear) in the DMA flexure test, the consistency in the data indicates that a high level of accuracy was exhibited in the test results. The findings of this study were published in a paper entitled "Accelerated Testing of Polymeric Composites Using the Dynamic Mechanical Analyzer", and presented at the American Society of Composites' 16<sup>th</sup> Technical Conference.

The second task was to develop a procedure to reduce the effect of physical aging during creep testing and to compare the analytical equations of creep compliance obtained from the master curve with the experimental data. Two material systems were used in this study: IM7/K3B and IM7/PETI-5. To reduce the physical aging effect, each material was subjected to a creep and recovery cycle and then rejuvenated before being tested at the next (higher) temperature. The rejuvenation process consisted of subjecting specimens to a temperature approximately 15° C above their glass-transition temperature, without applying any load, for a half hour after each creep cycle. Rejuvenation allows the free volume in the polymer to recover to its equilibrium state and effectively resets the aging clock. The advantage of this procedure is to eliminate the effect of the accelerated testing introduced by the high temperature on the material, simplify the analytical procedure to obtain the master curves, and obtain accurate results by using the same specimen for all creep-recovery cycles.

The results of this investigation show that the predicted creep compliance curves match the experimental compliance curves closely at temperatures at or below the selected reference temperature, but show slight discrepancies at temperatures above the reference temperatures. For best results a reference temperature should be selected at the application temperature or at a temperature higher than the application temperature of the material. A comparison between the creep properties of the two materials indicates higher creep compliance and creep rate in the IM7/K3B master curves as compared to the IM7/PETI-5 material. It appears that the IM7/PETI-5 material shows lower creep compliance and responds better to high temperature applications. The verification of the creep compliance equations obtained from TTSP theory was tested in this study. Samples of the results are shown in Figures 1 and 2 below.

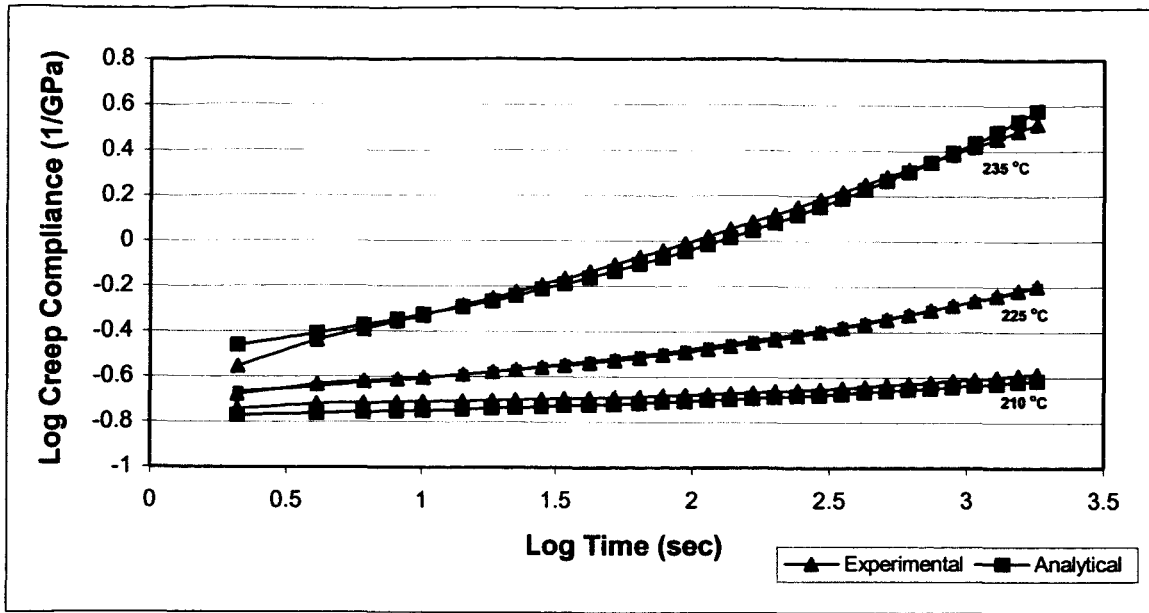


Figure 1. IM7/K3B Creep Compliance Master Curves: Analytical vs. Experimental Results

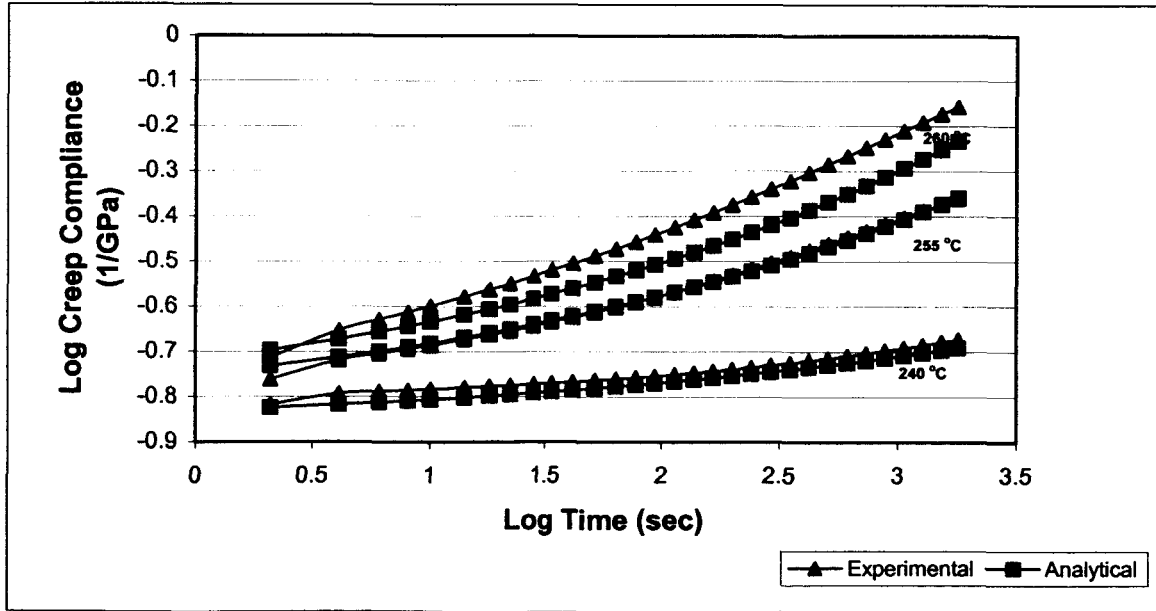


Figure 2. IM7/PETI-5 Creep Compliance Master Curves: Analytical vs. Experimental Results

The findings of this study were published in two papers. The first paper, entitled "Prediction of Long-term Creep Properties of Polymer Matrix Composites" was published in the proceedings of and presented at the ASC 18<sup>th</sup> Technical Conference, and the second was a student paper that was published in the College of Science and Engineering Journal at Winona State University.

The third task was to perform accelerated DMA creep tests taking into account the aging effects and compare the results with results from a previous investigation that consisted of tensile and compression tests. The same IM7/K3B and IM7/PETI-5 materials were used in this study. Work on this task is in progress, and preliminary results show that the long term creep compliance predicted by the DMA tests is higher than that predicted by tensile creep tests. Samples of these results are shown in Figures 3 and 4. However, no conclusions have been drawn from this investigation as it is still in progress; and the findings will be published in a paper to be submitted to the Journal of Composite Materials.

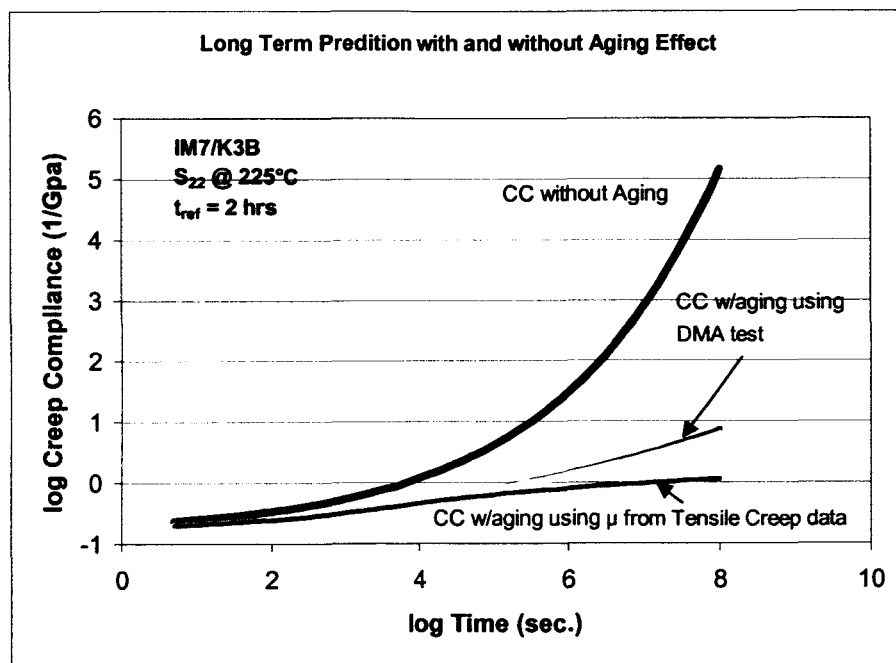


Figure 3. Long Term Prediction of Compliance for IM7/K3B Material

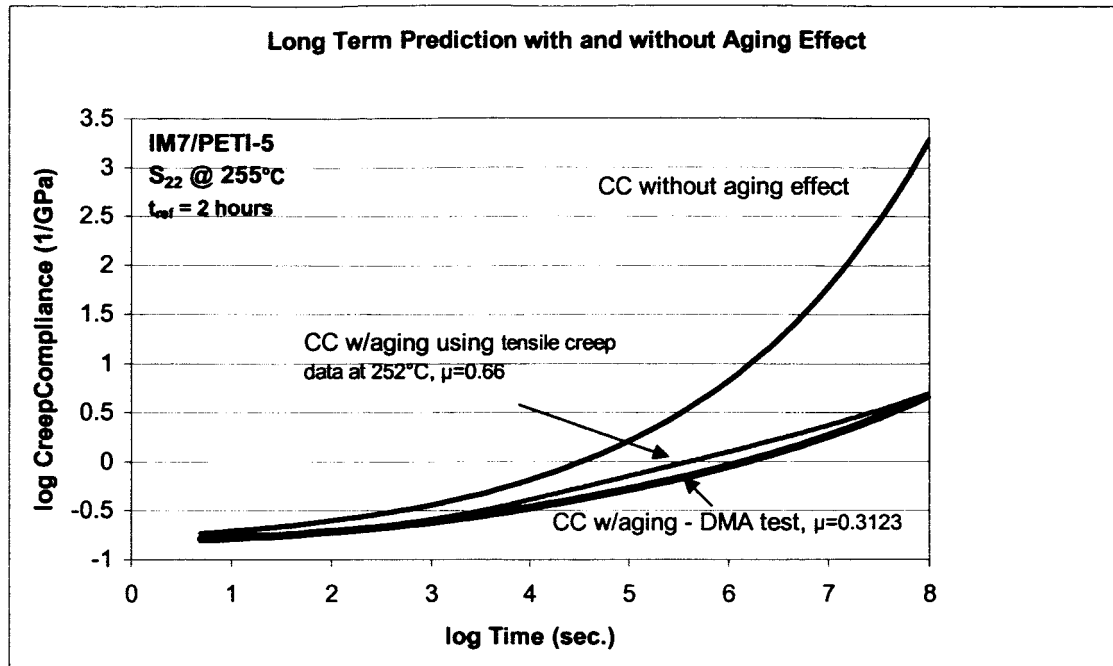


Figure 4. Long Term Prediction of Compliance IM7/PETI-5 Material

## CONCLUSIONS

A preliminary study of the long term creep behavior of polymer matrix composites shows that results obtained by DMA accelerated testing are similar to the behavior predicted by tensile and compression creep tests. A procedure developed to reduce the physical aging effects during creep testing proved effective in obtaining quick results for material selection purposes. However for accurate prediction of long term creep compliance, physical aging effects should be included in the experimental and analytical procedure.

Preliminary results of long term creep behavior and aging effects predicted by accelerated DMA flexure testing are slightly different from behavior predicted by tensile creep testing. Further study is being conducted to investigate the difference in these results and to analyze the effect of loading mode and size of specimens on the predicted long term properties.

Funding is being sought to complete the remaining tasks of the research project. These include correlation of frequency test data with creep test data to develop accelerated test method using frequency test procedure to find creep compliance, and development of a model to predict viscoelastic creep properties from accelerated frequency tests.

## **Publications**

The findings of the research project were published in the following three papers, and the results of the on-going investigation will be published in the fourth paper:

- [1] Abdel-Magid, B., and Gates, T., (2001), "Accelerated Testing of Polymeric Composites using the Dynamic Mechanical Analyzer," Proceedings of The American Society of Composites, 16<sup>th</sup> Technical Conference, September 2001.
- [2] Abdel-Magid, B., Gates, T., and Pawlitzke, B. (2003), "Prediction of Long-term Creep Properties of Polymer Matrix Composites," Proceedings of the 18<sup>th</sup> Annual Technical Conference of the American Society of Composites, October 2003.
- [3] Brad Pawlitzke and Beckry Abdel-Magid, (2003), "Accelerated Testing to Obtain Viscoelastic Creep Properties of Polymer Matrix Composites", Floruit-Journal of the College of Science and Engineering, Winona State University, Volume 01, No. 01, Fall 2003
- [4] Abdel-Magid, B., Gates, T., and Zanzmiller, J., "Testing Method and Scale-up Effects on the Viscoelastic Creep Properties of Polymeric Composites", in preparation to be submitted to the Journal of Composite Materials.